

**NOTE:** Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of  $\geq 0.01$  g if weighed in the laboratory or  $\geq 0.2$  g if weighed in the field) should not be used to collect samples.

### Sample Collection

Regardless of which sampling option is used (i.e., in-field methanol preservation with fill-line, EnCore™ sampler/sealed tube sampling/storage device with laboratory preservation, or in-field methanol preservation with field weighing) the desired ratio of methanol-to-soil should be 1-ml methanol to 1-gram soil,  $\pm 25\%$ . A soil sample with a minimum weight of 15 grams is required. The exact weight of the soil sample and the volume of methanol must be determined by the laboratory when calculating and reporting soil concentration data.

**NOTE:** The 1:1 soil-to-solvent ratio appears to work well for solid samples (e.g., sandy soil) that do not expand to soak up the methanol when it is added. On the other hand, many samples, such as those with a high organic content, may expand and soak up all the methanol, making it impossible to remove the methanol extract from the sample container for purging purposes. If the solvent does not cover all of the soil, volatile analytes will escape into the headspace and not be captured in the aliquot of solvent removed from the vial for analysis. **In all cases, the soil sample in the vial must be completely covered by methanol.**

Step 1: Obtain labeled and pre-weighed 40-mL or 60-mL VOA vials with open top screw caps that have Teflon-coated septa. Each vial should contain 15 mL or 25 mL of **purge-and-trap grade methanol** respectively. (Collect 15.0 grams of soil into a 40-mL vial, or 25.0 grams into a 60-mL vial.) Obtain enough to collect duplicate samples, a methanol trip blank, and an empty container per location to collect soil for calculating moisture content and dry weight.

Step 2: Transport the pre-labeled and weighed vials that contain the appropriate volume of methanol to the field in a cooler, which maintains a temperature of 4 degrees Celsius. Take precautions to avoid exposing the vials to exhaust fumes or other known airborne contaminants at all times. Use disposable gloves while collecting samples.

Step 3: Calibrate a field electronic balance to weigh 15.0 grams of soil, to the nearest 0.01 gram, for a 40-mL vial. The calibration check weight should approximate the expected combined weight of the closed sample vial containing methanol and the soil sample. Use a 10-30 mL disposable plastic syringe with the end cut off, or a special soil sampler, to obtain an undisturbed soil sample from freshly exposed soils. Collect soil samples within a few minutes, at most, from the time when the surface of the soil has been exposed to the atmosphere.

**NOTE:** Collect trial samples with the syringe, weigh and note the length of the soil column in the syringe to determine the length of soil corresponding to  $15.0 \pm 0.5$  grams. Discard all trial samples.

Step 4: Collect a soil sample, open a sample vial, and immediately extrude the soil sample into the vial carefully to avoid splashing methanol out of the container. Use a clean brush or paper towel to quickly brush any soil off the vial threads, then immediately seal the vial with the septum and screw cap. Wipe off any soil adhering to the outside of the vial. Gently swirl the sample to break up any soil clumps, if necessary, but do so only until the soil is covered with methanol. **The soil sample must be completely covered by methanol.**